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The Crystal Structure of 2-Hydroxy-N-[3(5)-pyrazolyl]-1,4-naphtoquinone-4-imine.

S. Pagola (Nat. Univ. Córdoba), S.L. Cuffini (CEPROCOR) and P.W. Stephens (SUNY at Stony Brook).

Beamline(s): X3B1

Introduction: The compound 2-Hydroxy-N-[3(5)-pyrazolyl]-1,4-naphtoquinone-4-imine is a new potential cytotoxic and anti-*Trypanosome-Cruzi* agent. The molecule is shown in figure 1. This compound presents three polymorphs. The crystal structure of one of them, pure and not solvated, has been solved by high resolution X-ray powder diffraction data.

Methods and Materials: The diffraction pattern has been taken at the X3B1 beamline. It has been indexed using the program ITO. The monoclinic unit cell (after Rietveld refinement) is: a= 18.4437(1) Ă, b=3.99677(2) Ă, c=14.5304(1) Ă, α=90°, β=102.7086(6)°, γ=90°. The space group $P2_1/c$ has been determined from the observation of the systematic absences. An estimation of density suggested Z=4. The Le Bail fit has been done with the program GSAS, Rwp=7.26%.

The crystal structure has been solved with the program PSSP [1], using the first 50 integrated intensities. The molecule to locate has one internal degree of freedom, thus 7 structural parameters had to be determined. The structure solution and final Rietveld refinement were tried with the molecular geometry optimized by semiempirical methods and also with the geometries of both cycles taken from entries of Cambridge Structural Database (CSD). In the last case, 12 parameters were used for the structure solution and both cycles were located independently.

Results: A representation of the crystal structure along the [010] direction is shown in figure 2. The final Rietveld refinement was done with the program GSAS, using two rigid bodies to refine the position and orientation of both rings, without angle or bond distance constrains. The plot is shown in figure 3. Rwp=7.52%.

The position of hydrogen atoms were included in the final Rietveld refinement (as they were determined in the structures of both CSD entries), except the position of the hydrogen in the hydroxyl group, for which the occupation of the oxygen atom was refined instead.

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References: [1]. Program PSSP. Available at http://powder.physics.sunysb.edu.

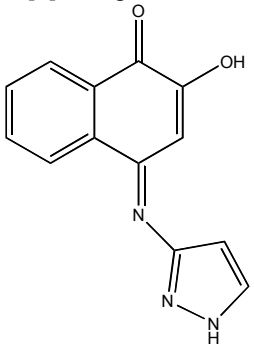

Figure 1: The molecule 2-Hydroxy-N-[3(5)-pyrazolyl]-1,4-naphtoquinone-4-imine

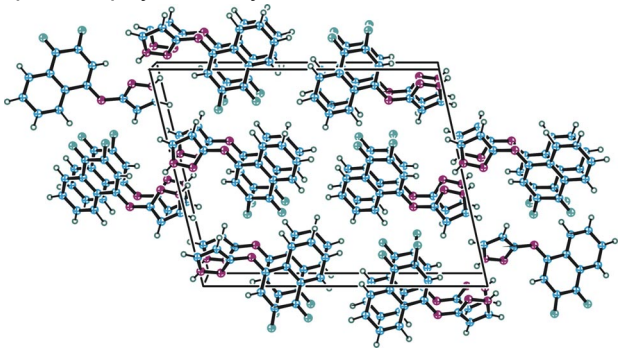

Figure 2: The crystal structure. The position of the H atom of the hydroxyl group was not determined.

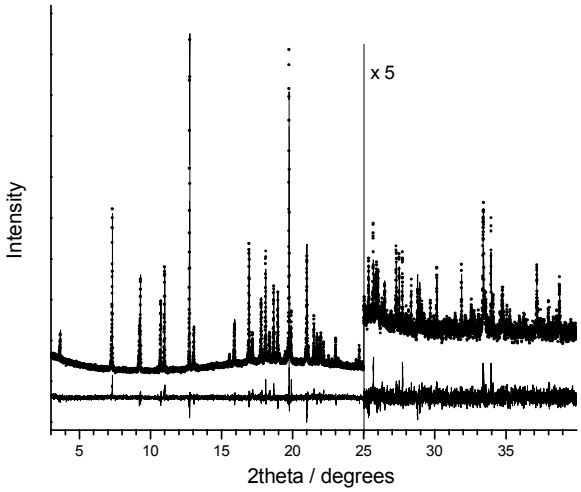

Figure 3: Plot of the final Rietveld refinement.